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PATENT & TRADEMARK OFFICE

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09/831,724	12/8/2000	124-852	Marie Yamnitzky

Evidentiary Declaration under 37 C.F.R. §1.132

I, Ian Charles Sage, a British subject, do hereby declare as follows:

1. That I hold the degrees of BSc with Honours and PhD from the University of Durham.

That I am a Fellow of the Royal Society of Chemistry and a Chartered Chemist.

That since June 1992 I have been employed at QinetiQ (previously DRA/DERA) in Malvern, where I hold the position of QinetiQ Fellow. Since 1992, I have been responsible for technical direction and programme delivery in a variety of technical areas including:

- Invention and development of novel liquid crystal materials and devices.
- Development of liquid crystal polymer materials and devices.
- Specification and evaluation of liquid crystal displays, and consultancy concerning their use.
- Non-display applications of liquid crystal technology.
- Development of large area spatial light modulator technology
- Applications of organic semiconductor technology, including light emitting and non-emissive applications of both polymers and low molecular mass organic semiconductors.
- Invention, development and exploitation of a novel damage sensing technology for composite structures exploiting triboluminescent sensors.
- Development of novel UV curing polymers as specialist adhesives and coatings.
- Investigation of materials and techniques for surface alignment of liquid crystals

That from 1976 to May 1992 I was employed by Merck Ltd (previously BDH Ltd). In 1986 I was appointed Organic Development Manager with overall responsibility for new product introductions based on organic chemistry, and reporting to the Development Director. From the mid 1980s, I led BDH/Merck input into a series of major collaborative projects, with electronics and device based companies and universities as partners. These included:

- Organic compounds for optical and non-linear optic applications
- Anisotropic fluorophors for liquid crystal displays
- Long wavelength fluorophors for display applications (Project co-ordinator)
- Liquid crystal switching elements for integrated optics
- Liquid crystal display on a silicon active matrix
- Large format storage display using ferroelectric liquid crystals

From 1987, I was also responsible for the development of formulations and coating technologies for thermochromic materials. This work included formulation of surface coatings for screen print, gravure, inkjet, transfer and hot press processes. I was also responsible for the formulation of compositions for supply to the cosmetics industry for use in skin care products.

During my time at Merck I became a world authority in the field of liquid crystal technology. Principal technical achievements included the introduction of fluorination as a route to ferroelectric liquid crystals, development of the first LCs compatible with evanescent wave switching in a silica waveguide, and development of practical routes to the ultrapurification of materials for non-linear optic applications. During that period I was responsible for development of a wide range of liquid crystal materials, both single compounds and ready for use mixtures. I worked closely with liquid crystal display manufacturers throughout the world to improve display performance by providing optimised liquid crystals and a number of the mixtures developed by my group became industry standard materials.

That I hold/have held the following offices:

- Present committee member and Chair, UK chapter, Society for Information Display
- Programme chair, Eurodisplay 2005
- Committee member, Royal Society of Chemistry, Materials Chemistry Forum
- Member, Engineering and Physical Science Research Council peer review college.
- Member of Editorial Board, Journal of Materials Chemistry (2000-2002)
- Member of organising committee, International Liquid Crystal Conference, York, 1984
- Member of SERC Molecular Electronics Committee, 1987-1989
- Four terms as committee member and two as membership secretary of the British Liquid Crystal Society
- Member of organising committee, International Conference on Ferroelectric Liquid Crystals, Cambridge, 1995
- Session chair, International Conference on Ferroelectric Liquid Crystals, Cambridge, 1995
- Member of Programme Committee, Eurodisplay, Birmingham, 1996
- Session chair, Eurodisplay, Birmingham, 1996
- Session chair, EID, 1997

That I am the author or co-author of the following patents, publications and conference papers:

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M Verrall, D Beattie, D Coates, I Sage and K Lymer, "Ferroelectric switching liquid crystal polyacrylates-the control and effect of molecular weight" *Ferroelectrics*, **181**, 327, (1996)

J Ebbutt, R M Richardson, L Taylor, K M Blackwood, I C Sage, D R Beattie and M Verrall, "Layer structure in processed ferroelectric sidechain liquid crystal polymers", *Ferroelectrics*, **181**, 261, (1996)

A W Hall, D Lacey, J S Hill, K M Blackwood, M Jones, D McDonnell and I C Sage "Synthesis and evaluation of a series of novel 2-substituted poly(allyl alcohol) sidechain liquid crystalline oligomers exhibiting ferroelectricity" *Liq Cryst*, **20**, 437, (1996)

G P Bryan-Brown and I C Sage, "Photoinduced ordering and alignment properties of poly(vinyl cinnamates)" *Liq Cryst*, **20**, 825, (1996)

G P Bryan-Brown, C V Brown, J C Jones, E L Wood, I C Sage, P Brett and J Rudin, "Grating Aligned Bistable Nematic Device" *SID Digest*, **28**, 37, (1997)

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I C Sage, D G McDonnell, J C Jones and A Slaney, "Ferroelectric liquid crystal devices", PCT 95 16760 (51pp)

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G P Bryan-Brown, I C Sage and E L Wood, "Liquid Crystal Device", GB Pat 9713164.3

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C.W.Slinger, P.Brett, V.Hui, G.Monnington, D.Pain, I.Sage "Optimisation strategies for MACH - a novel, electrically controllable, computer generated hologram" SPIE Photonics West 1997, San Jose, February, 1997 (Invited paper)

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Hong Kong, 1982
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Peking, Shanghai, 1985
Changsha, 1987
Tokyo, Osaka, 1988
Shenzhen, 1989
New York, 1989
Tokyo, Osaka, Seoul, 1990
New York, 1990

I further declare that:

1. I am an inventor in and am familiar with U.S. Patent Application No 09/831,724 to Wood *et al* based on application no. 9826845.1, filed in the United Kingdom on 8th December 1998 (hereinafter "the Application").
   QinetiQ Ltd is the assignee in respect of the Application.

2. The Application describes and claims an organic light emitting diode device comprising a substrate bearing an organic layer sandwiched between electrode structures, wherein the organic layer comprises a hole transporter, an electron transporter and a light emitter wherein either or both of the electron transporter and light emitter comprise a material according to formula 1, characterised in that the organic layer is a single layer.

3. The materials are of the general formula $F_2B(A)(D)$, where A and D are independently selected N, O and comprise part of a number of larger ring systems as described.

4. Japanese Patent No. 9-289081 ("Hiroyuki") describes an organic light emitting diode in at least two discrete layers, using boron di-fluoride compounds. The ligands which are claimed in the Application are however, clearly differentiated from the materials which are disclosed in the Hiroyuki patent and hence the use of such compounds as claimed in the Application are novel.

5. The Hiroyuki patent describes in the paragraph bridging page 10 and 11, that '...In the organic electroluminescent element of the present invention, at least one among the hole transporting layer 3 and organic electron transporting layer 4 must also function as the light emitting layer....' Contrary to the examiner's comments that Hiroyuki has disclosed at least one layer containing all three species, electron/hole transporters and light emitters, there is no suggestion in the Hiroyuki patent that the BF2 complex will behave as both a light emitter and electron transporter. Additionally, the Hiroyuki patent clearly describes at least two physical layers between the electrodes, these being layers 3 and 4, whereas the Application clearly describes at only one layer.

6. Further, Hiroyuki did not suggest that a device may be manufactured with only the boron difluoride as the electron transporting compound. Additionally the prior art cited by the examiner does not allude to the light emitter and the electron transporter being the same compound, whereas this is a feature of our invention as claimed.

7. US Patent No. 5,281,489 ("Mori") describes an organic light emitting diode which is manufactured in a single layer, however the patent does not use boron difluoride compounds, instead it discloses the use of a different class of compound as compared with the Application.

8. The examiner has expressed a view that, although the use of the compounds in a device satisfies the novelty requirement, the claimed compounds are sufficiently similar in structure to the compounds disclosed in "Hiroyuki" such that when the latter is read in combination with the teachings of "Mori", that is single layer technology, it would be obvious to incorporate the boron difluoride compounds used in our invention into a single layer. However it has been shown by the International Preliminary Examiner that the authors of citation D1 (Proceedings 1997 ACS San Francisco Vol. 38, No. 1, pages 339-340), which describes a pyrromethane-BF2 complex, were not able to produce a functioning organic LED containing only one layer. Consequently in my judgement it would be incorrect to assume that boron difluoride compounds according to the Application could be successfully incorporated into a single layer.

9. There is a trade off between single and multiple layered devices; while the deposition of separate layers is costly, time consuming and difficult to control, two

layer devices are known to be efficient in both energy and light output, which makes them a desirable product. Indeed, devices using double layer structures were introduced to the art by Tang and VanSlyke (Applied Physics Letters 51(12), 913 (1987)) specifically because this structure promoted high efficiency. To quote from their abstract, "emission... confined near the organic interface region. High external efficiency...and brightness are achievable" The organic interface region is essential to the improvements in Tang and Van Slyke's device, and for this interface to exist, a double (or multiple) layer structure is essential. The production of single layer devices, which are cheaper to produce but in general have lower efficiencies, has been disclosed in many documents. Achieving efficient devices from a single layer structure which is convenient and inexpensive to fabricate has been an outstanding problem, which our invention has overcome. There is no indication whatsoever in the prior art to suggest that use of our specific boron compounds will lead to an increase in efficiency of a single layer device.

10. However, it is the case in this field that the correct selection of the light emitting structure can bring about significant changes in the emitting properties and behaviour of such devices and in my judgement the use of the compounds in a device as claimed does in fact exhibit significant, advantageous and unexpected differences over the prior art devices shown in Hiroyuki.

11. To demonstrate the unexpected efficiency of the boron difluoride compounds, as used in devices according to the present invention an experiment was set up to compare the efficiencies of the Mori single organic phase and the single phase according to the Application. A solution was prepared containing 0.4g of N,N'-diphenyl-N,N'-di-3-tolylbenzidine, 0.4g of 2-(4-t-butylphenyl)-5-biphenylyl-1,3,4-oxadiazole and 0.4g of poly-N-vinylcarbazole in 13.0g of 1,2-dichlorobenzene. The solution was left to stir on a heated plate until the solids had fully dissolved, then divided in half. To one half was added 0.00016g of 1,3,5,7,8-pentamethyl-2,6-di-n-butylpyrromethene-difluoroborate, used to provide devices according to the present Application. To the other half was added 0.00016g of the laser dye coumarin 6, which is a known luminescent dopant for organic electroluminescent devices according to Mori.

12. Pieces of 2 inch square ITO coated glass (100Ω/□) were cleaned with acetone and propan-2-ol, then spin coated with one or other of the above solutions at 1000rpm

for 30 seconds. The coated glass was transferred to a heated plate at 90°C to evaporate solvent and left to dry for 5 minutes, then heated at 90°C in an oven for 30 minutes.

13. The coated plates were transferred to a vacuum deposition apparatus and at a pressure of 10^{-6} millibar magnesium (1000A) followed by silver (1000A) were evaporated onto the polymer through a perforated metal mask from thermal sources to form an array of circular metallic contacts. Each contact defined an organic light emitting device. After coating, the devices were removed and electrical contact made to each ITO glass substrate by means of a wire attached by indium solder.

14. Electrical contact was made to the magnesium/silver electrodes from the negative terminal of a current source and a current of 100.00mA was passed through each device tested. Light emission could be observed from the ITO glass side of the devices. The brightness of each of the devices was measured under identical current drive conditions using a Photo Research type 714 spot photometer, and results quoted are the average of those measured for several devices.

15. The brightness measured for the device comprising a layer doped with coumarin 6, by Mori, was 187cd/m^2. The brightness measured for the device according to our Application, which used 1,3,5,7,8-pentamethyl-2,6-di-n-butylpyrromethene-difluoroborate, however, was 281cd/m^2. Therefore the device made according to the present Application achieved a brightness which is 50% greater than that made according to Mori. Since the drive current and voltage were the same, the device also achieved a power efficiency 50% higher than the device made according to compounds stated in Mori.

16. There is no indication in any of the prior art that such unexpected benefit can be achieved in devices by using the compositions in our Application; prior art using similar luminescent materials in different device structures merely note the relatively narrow emission spectrum. In fact the device structures revealed in the prior art are not such as to provide the benefits of improved brightness and efficiency from a simple and cheaply manufactured device.

17. Consequently I believe that the Application has the merit of for the first time making available to the field a single layer device incorporating a range of boron

difluoride compounds and organic LED devices incorporating the same which provide improved performance and a greater range of functionality. Moreover, no such advantages of single layer manufacture and dual functionality of the Boron diflouride compound are even hinted at in Hiroyuki and hence Hiroyuki should not be presented as providing a route to the new and improved devices which have been made available by the Application.

18. Consequently it may be seen that Hiroyuki gives no guidance to the skilled person that would point him in the direction of using the Boron diflouride compounds in this dual functionality which forms a part of the subject of the Application.

19. In summary therefore, based upon my experience in this field and the available information, I would state that the Application provides a novel and distinctive use of boron difluoride compounds, which is completely outside of the scope of the disclosure of Hiroyuki. The latter shows only very broadly similar materials and provides no teaching at all that could be said to guide the skilled person's efforts to provide a single layer device with efficiencies far exceeding known prior art single layer compositions. Such an effort to produce single layer devices could have taken many possible directions, indeed resulting in failure for certain cited prior art, hence the specific direction leading to applicants' invention must be regarded as being an unexpectedly meritorious choice.

20. I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the Application or any patent issuing thereon.

Date 2nd May 2003 ..... Ian Sage .....

Ian Charles Sage